AFRL-ML-WP-TP-2007-433

DETERMINATION OF THE MISORIENTATION DEPENDENCE OF THE RELATIVE GRAIN-BOUNDARY ENERGY OF COMMERCIAL-PURITY NICKEL (Preprint)

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FEBRUARY 2007

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MATERIALS AND MANUFACTURING DIRECTORATE
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AIR FORCE MATERIEL COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7750

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YY)*	2. REPORT TYPE	3. DATES COVERED *(From - To)*
February 2007	Journal Preprint	

4. TITLE AND SUBTITLE

DETERMINATION OF THE MISORIENTATION DEPENDENCE OF THE RELATIVE GRAIN-BOUNDARY ENERGY OF COMMERCIAL-PURITY NICKEL (Preprint)

5a. CONTRACT NUMBER
IN-HOUSE

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER
62102F

6. AUTHOR(S)

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5d. PROJECT NUMBER
4347

5e. TASK NUMBER
RG

5f. WORK UNIT NUMBER
M02R2000

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Metals Branch, Processing Section (AFRL/MLLMP)
Metals, Ceramics, and Nondestructive Evaluation Division
Materials and Manufacturing Directorate
Air Force Research Laboratory, Air Force Materiel Command
Wright-Patterson Air Force Base, OH 45433-7750

Wright State University

Carnegie Mellon University

8. PERFORMING ORGANIZATION REPORT NUMBER
AFRL-ML-WP-TP-2007-433

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Materials and Manufacturing Directorate
Air Force Research Laboratory
Air Force Materiel Command
Wright-Patterson AFB, OH 45433-7750

10. SPONSORING/MONITORING AGENCY ACRONYM(S)
AFRL-ML-WP

11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S)
AFRL-ML-WP-TP-2007-433

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES
PAO Case Number: AFRL/WS 07-0579, 15 March 2007. Submitted for publication to Scripta Materialia (Elsevier). The U.S. Government is joint author of this work and has the right to use, modify, reproduce, release, perform, display, or disclose the work.

14. ABSTRACT

Columnar-grained samples of commercial-purity nickel were analyzed to establish the relative energy of <001> tilt boundaries as a function of misorientation. For this purpose, an electron-backscatter-diffraction (EBSD) technique was used to determine the grain-boundary geometry and misorientations across grain boundaries at triple-junction locations in two-dimensional sections perpendicular to the sample fiber texture. Analysis of the triple-junction dihedral angles and misorientations revealed that the relative boundary energy followed a classical Read-Shockley dependence.

15. SUBJECT TERMS
Directional solidification, EBSD, Nickel, Grain boundary energy, Misorientation

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT:	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON (Monitor)
a. REPORT	b. ABSTRACT	c. THIS PAGE			David W. Mahaffey
Unclassified	Unclassified	Unclassified	SAR	12	19b. TELEPHONE NUMBER *(Include Area Code)* (937) 255-1305

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. Z39-18

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Abstract

Columnar-grained samples of commercial-purity nickel were analyzed to establish the relative energy of <001> tilt boundaries as a function of misorientation. For this purpose, an electron-backscatter-diffraction (EBSD) technique was used to determine the grain-boundary geometry and misorientations across grain boundaries at triple-junction locations in two-dimensional sections perpendicular to the sample fiber texture. Analysis of the triple-junction dihedral angles and misorientations revealed that the relative boundary energy followed a classical Read-Shockley dependence.

Keywords: Directional solidification; EBSD; Nickel; Grain boundary energy; Misorientation

Introduction

A variety of simulation methods for predicting microstructure evolution during processing and service are emerging as powerful tools for material designers. These tools include techniques for modeling phenomena such as grain growth, precipitation, recrystallization, and texture formation. For example, Monte-Carlo (MC) approaches for simulating grain growth have matured significantly during the last decade [1]. Key input data for MC models include the dependence of grain-boundary energy and mobility on grain-boundary character. Measurement of boundary properties is generally very difficult. Hence, relatively few measurements of this sort can be found in literature [2, 3]. To help overcome the challenges associated with these measurements, techniques such as molecular dynamics have been developed to predict boundary energy and mobility [4]. Nevertheless, such theoretical treatments still require experimental verification.

A number of methods have been developed to measure boundary energy and mobility in single-phase polycrystalline materials. These include techniques such as those based on thermal grooving, measurements of boundary motion, etc [5, 6, 7]. Recently, a novel technique was introduced by Adams, *et al*. [8]. This method is based on electron backscatter diffraction (EBSD) to determine boundary misorientation/geometry and is applicable to both two-dimensional (e.g., columnar) and three-dimensional (e.g., equiaxed) grain structures. In

essence, dihedral angles (between adjacent boundaries) and grain-boundary curvatures at triple junctions are analyzed in the context of Herring's treatment of boundary energy, or Young's equations if grain boundary character is simplified to depend on misorientation only. Yang, et al. demonstrated this approach using high-purity aluminum [9].

Nickel-base superalloys comprise a class of materials for which validated modeling techniques for grain growth would be especially useful. For example, a quantitative description of normal and abnormal grain growth during supersolvus heat treatment has been sought for many years in the aerospace industry. The present work was undertaken as a first step in meeting this need. Its objectives were to determine the relative energy of <001> tilt boundaries of unalloyed nickel and to compare the results to previous measurements in other materials and the Read-Shockley equation [10].

Materials and Procedures

Materials. A special lot of commercial-purity nickel (CP Ni) was melted and directionally solidified (DS) to produce 12 x 50 x 225 mm bars having a columnar grain structure (Figure 1) with a <001> fiber texture parallel to the long direction. Its measured composition (in weight percent) was Bi < 0.02, Pb < 0.02, Te < 0.02, Ti < 0.02, and balance Ni. The average grain diameter was approximately 1.5 mm as shown in the optical micrograph (Figure 1).

Experimental Procedures. The fiber-textured, columnar-grain structure of the program material enabled the analysis of 2-D sections for the determination of relative grain-boundary energy. For this purpose, slices measuring 3 to 5 mm thick were cut perpendicular to the fiber axis of the DS bars and prepared for EBSD examination using standard metallographic techniques. The samples were final polished on 0.06 μm colloidal silica and electropolished in a solution of 590 ml methanol, 350 ml 2-butoxy-ethanol, and 60 ml 60% concentration perchloric acid at 20V for 20 minutes.

EBSD was conducted in an FEI XL30 field-emission-gun scanning-electron microscope (FEG SEM) using TexSem Laboratories software version 4.0 [11]. EBSD scans focused on delineating the grain-boundary geometry and misorientation between grains at triple junctions. For this purpose, coarse scans with a step size of 25 μm were utilized to locate the triple points, and fine scans with a step size of 1 μm were used to measure the geometry and character of the triple junctions thus located. This procedure was used to characterize approximately 150 triple junctions.

Data Analysis. The EBSD data were analyzed to determine dihedral angles at the triple junctions and thus provide input for the Yang analysis of relative grain-boundary energy. The grain boundaries established via EBSD were first highlighted by a thresholding-and-cropping technique [12]. The thresholding process eliminated grey-scale noise in the images. Subsequently, excess grain-boundary area was cropped from each image. The skeletonized microstructures so produced were input to special software [13] to determine the inclination angle of each grain boundary and hence the associated dihedral angles at each triple junction. The columnar nature of the microstructure

indicated that the angles measured on the polished sections were indeed true dihedral angles; serial sectioning would, of course, be required for typical three dimensional microstructures. The data were then input to a Microsoft Excel™ spreadsheet listing the dihedral angles and misorientations characterizing the grain boundaries at each triple junction.

By neglecting the variation in boundary energy with boundary normal, the analysis of the spreadsheet data could be based on Young's relation [10] between the measured dihedral angles (χ_1, χ_2, χ_3) and the corresponding relative boundary energies (γ_1, γ_2, γ_3) at triple junctions (Figure 2), viz. :

$$\frac{\gamma_1}{\sin\chi_1} = \frac{\gamma_2}{\sin\chi_2} = \frac{\gamma_3}{\sin\chi_3}$$

The data for each triple junction were sorted into misorientation bins using the procedure developed by Yang and his co-workers [8, 13]. Specifically, the misorientations were sorted into one of 14 bins each of whose width was between 2 and 5 degrees, thus spanning the total misorientation range between 0 and 45 degrees possible for cubic metals with a <001> fiber texture. The minimum bin size of 2 degrees was chosen in view of the resolution limit of the EBSD technique. The relative boundary energy as a function of misorientation was determined by a statistical multiscale analysis of the binned data [14]; i.e., a method of finding the variation of boundary energy with misorientation that most nearly satisfies Young's equation at each triple junction. The results were then compared to the Read-Shockley equation for the energy of low-angle grain boundaries.

Results and Discussion

EBSD-determined pole-figure results (Figure 3) indicated that the texture of the CP Ni program material was close to, but not exactly, a <001> fiber texture. For example, the finite width of the central peak in the 001 pole figure indicated some deviation from the ideal fiber texture. The individual EBSD scans can only cover a limited-size grain-sampling area, thus explaining the incomplete nature of the <001> fiber texture. Nevertheless, the grain-boundary misorientations were always between 2 and 60 degrees. More specifically, approximately 95% of the misorientations were between 2 and 45 degrees. For a more quantitative insight, the deviation from the <001> fiber texture was estimated by first finding the axis-angle pair representing the misorientation between two adjacent grains and then calculating the angle between that axis and the [001] of the corresponding grains. The maximum deviation from <001> fiber texture was thus found to be less than 4 degrees with an average of approximately 2 degrees (Figure 4).

The misorientation distribution expected for a perfectly-random <001> fiber texture would exhibit a uniform density over the range 0-45°. The CP Ni characterized in this work showed a misorientation distribution that was sufficiently close to this limiting case to justify the application of the single misorientation parameter Yang analysis for the present data set (Figure 5). The

experimentally-determined dependence of relative grain-boundary energy on misorientation for CP Ni is shown in Figure 6. These results revealed a monotonic increase in energy with misorientation (θ) from 0 to ~15 degrees. Furthermore, the boundary energy appeared to be approximately independent of misorientation for θ > 15 degrees, except for a weak local minimum at θ ~ 30 degrees. This misorientation is close to the Σ17a <001> tilt boundary [15]. However, the sparseness of the data set and the chosen bin size precluded a definitive conclusion regarding the nature of the relative boundary energy at this misorientation. In general terms, the absence of a marked variation in the grain boundary energy beyond the Read-Shockley regime is in good agreement with measurements for other fcc metals such as Al [16].

The experimental results were also compared to the Read-Shockley equation for the relative energy (γ) of low angle boundaries, i.e.,

$$\gamma = \gamma_m \frac{\theta}{\theta_m}\left(1 - \ln\frac{\theta}{\theta_m}\right)$$

in which γ_m denotes the energy of a high angle boundary, and θ_m = 15 degrees. This comparison (Figure 6) showed excellent agreement between the measured values and Read-Shockley model.

Summary and Conclusions

The Yang, et al. approach for estimating the misorientation dependence of the relative grain-boundary energy in polycrystalline materials was applied to commercial-purity nickel samples having a columnar grain structure and <001> fiber texture. The data revealed a typical dependence of relative energy on misorientation (θ); i.e., an increase of energy with misorientation for $0 \leq \theta \leq 15$ degrees and approximately constant energy for θ > 15 degrees. The experimentally determined energy dependence on misorientation showed excellent agreement with the predictions of the Read-Shockley model.

The results of this investigation demonstrated the applicability of the Yang approach for the determination of the misorientation dependence of the relative boundary energy for CP Ni and revealed that the measured dependence followed the Read-Shockley equation.

Acknowledgements

This work was conducted as part of the in-house research of the Metals Processing Group of the Air Force Research Laboratory's Materials and Manufacturing Directorate. The support and encouragement of the laboratory management and the Air Force Office of Scientific Research (Dr. C. S. Hartley, program manager) are gratefully acknowledged. The authors would like to thank Dr. M. Glavicic and Dr. A. Salem for technical discussions and Ms. T. Bennett and Mr. J. Miller for assistance with analysis. Two of the authors were supported under the auspices of AF Contracts F33615-02-2-5800 (AES) and F33615-01-2-5225 (ADR).

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Figure Captions

Fig 1. Optical macrograph of the columnar-grain structure in directionally-solidified commercial-purity nickel samples.

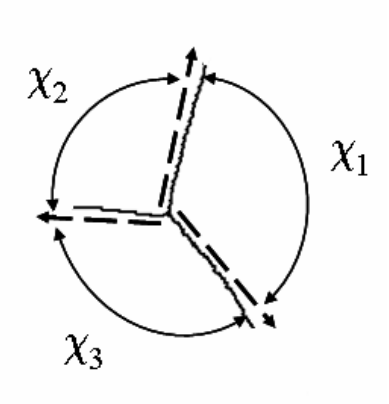
Fig 2. Schematic representation of the relation between the relative grain-boundary energies and the corresponding dihedral angles at a triple junction.

Fig 3. <001> pole figure derived from EBSD data; the z-direction is at the center of the pole figure.

Fig 4. Calculated deviation of the misorientation axis from the [001] crystal direction.

Fig 5. EBSD-determined misorientation distribution for commercial-purity nickel.

Fig 6. Comparison of the experimental dependence of the relative boundary energy on misorientation for commercial-purity nickel (points) and the trend predicted by the Read-Shockley equation (smooth curve).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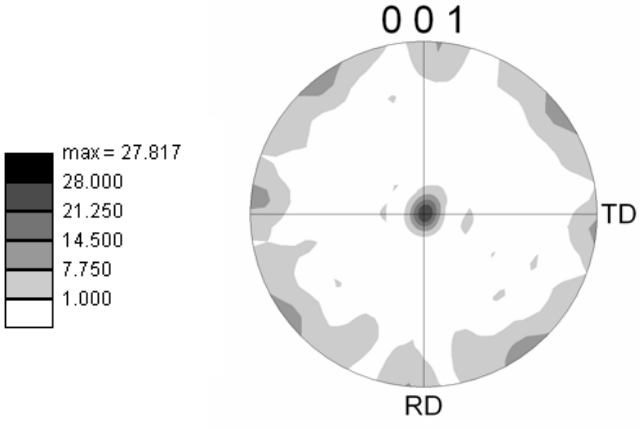
χ_1
χ_3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TD
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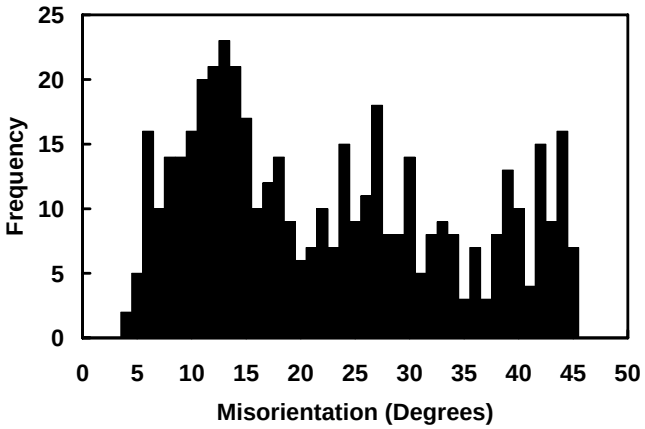
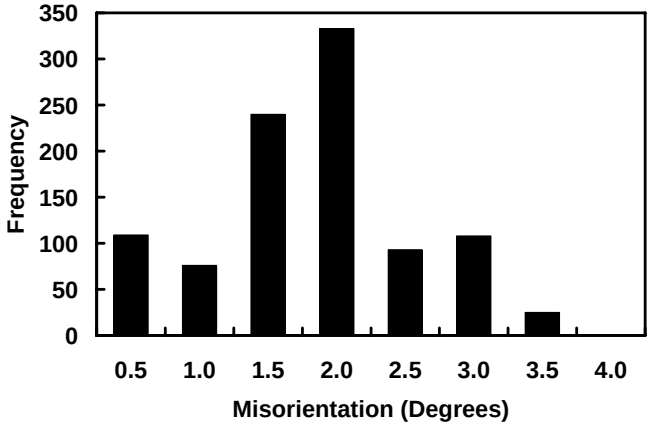

Frequency
Misorientation (Degrees)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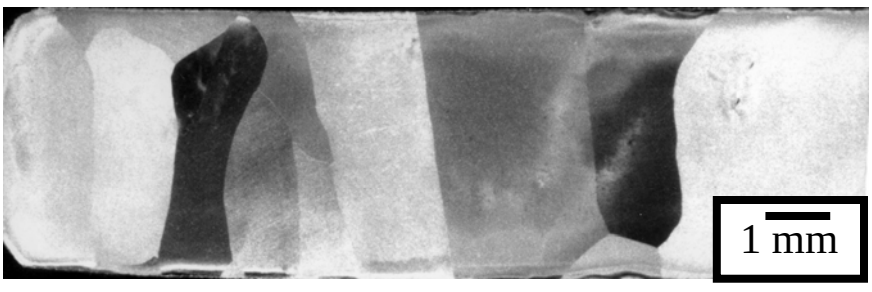
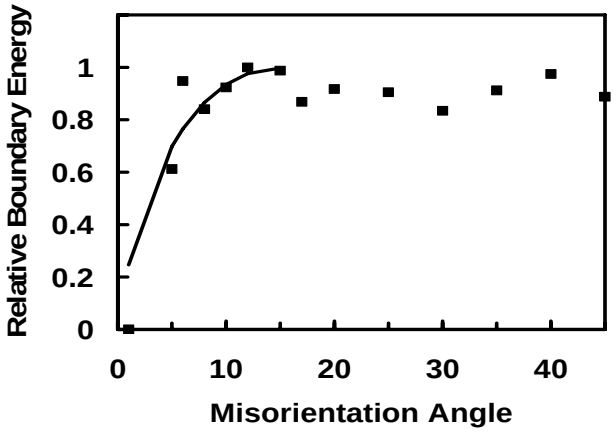

1 mm


Relative Boundary Energy
Misorientation Angle